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Mechanical performance of PEEK produced by additive manufacturing

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Abstract

Recent developments in production methods for polymeric materials have meant that thermoplastics for high temperature mechanical performance can now be selectively laser sintered. This paper describes the performance and the potential applications of EOS PEEK HP3: a high temperature, laser sintered thermoplastic material.

Thermal, tensile, flexural, compressive and fractural tests were conducted to assess the mechanical response of the material. Physical properties, such as porosity and roughness are also presented along with a discussion on the failure mechanisms of the material during testing.

Finally, the significance of this material in the production of prototype parts, the mechanical requirements of the polymer and limitations of its applications are outlined.

Key words: Additive manufacture, selective laser sintering, Polyether ketone, high temperature

Research highlights

- The mechanical properties of sintered PEEK are similar to injection moulded PEEK
- The mechanical properties of sintered PEEK show an improvement over sintered PA12
- Failures of laser sintered PEEK are predominantly brittle in nature
- Partial sintering between manufactured layers limit structural strength of the material
- Small scale porosity apparent in sub surface layers of sintered PEEK

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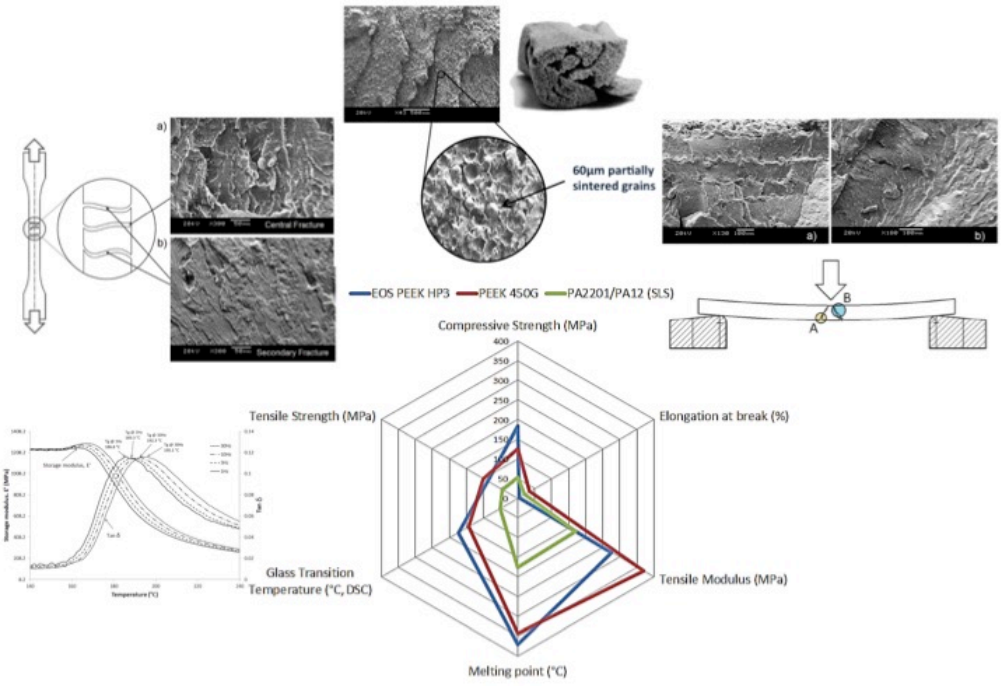
Graphical Abstract



1.0. Introduction

Recent developments in processing methods of polymeric materials have meant that high temperature polymers for structural and mechanical applications can now be selectively laser sintered. High temperature selective laser sintering (HT-SLS) is a method of additive manufacturing used mainly in the aerospace and medical industries (1). However, the use of this process in the development of mechanical components for dynamic applications is receiving significant interest (2).

Additive manufacturing (AM) allows for a significant improvement in design flexibility; high-complexity bespoke parts can be produced economically, without the need for expensive tooling. There are a number of advantages that SLS has over other polymer AM methods, these include the elimination of the need for support structures when overhangs and thin-walled sections are incorporated into designs, the elimination binding agents and the large range of potential polymer materials that can be processed via SLS, relative to other AM techniques (3) . Currently, there is a growing number of applications for bespoke components which can withstand high mechanical loads, are biocompatible and can tolerate high-temperature operation. High performance thermoplastics such as polyamide 12 (PA12) have been used in additive manufacturing for several years to produce dense parts with relatively high mechanical strength (4–8). However, certain material characteristics, such as relatively low melting temperatures and low glass transition temperatures, limit their application. Therefore, there has been an increasing interest in producing high temperature materials for use in high-temperature selective laser sintering.

EOS PEEK HP3 is a material belonging to the group of poly-aryl-ether-ketones (PAEK). This group of polymers have been shown to demonstrate superior performance characteristics compared to other engineering polymers (9,10). EOS PEEK HP3 semi-crystalline, thermoplastic material was developed by Electro Optical Systems (EOS GmbH., Munich, Germany) and Victrex (Victrex plc., Lancashire, UK) for use on the high-temperature EOSINT P800 machine. The EOSINT P800 high temperature system uses a CO_2 laser that can run at temperatures of 385°C to build three-dimensional geometry in 0.1 mm layers. This process is capable of laser-sintering high-performance polymers such as polyetherketone (PEK) that otherwise could not be manufactured using conventional laser-sintering systems.

There is very little information available on the properties of EOS PEEK HP3. The majority of the data are based on manufacturer's values, where their significance for a given application is not immediately apparent (EOS GmbH, 2014). Greses and Stoko, and Schmidt *et al*. outlined the selective laser-sintering (SLS) process for high performance polymers although there was limited analysis of the material properties (12,13). Beard *et al*. have attempted to characterise EOS PEEK HP3 components; however, their study was limited to considering only a few practical properties (14).

This paper outlines the physical and mechanical properties of EOS PEEK HP3. Characterisation of the tensile, flexural, compressive, fracture and thermal properties was performed, together with specific physical material properties such as porosity and roughness. This highlights the potential of EOS PEEK HP3 as a material for high performance applications.

2.0. Test Methodology

Test samples were manufactured by 3D Systems Corporation (Langhorne, PA, USA), using an EOSINT P800 (EOS Electro Optical Systems 2011a) high-temperature laser-sintering system. EOS PEEK HP3 powder, specifically designed for the EOSINT P800 additive manufacturing system, was used to produce the parts. This is shown schematically in Figure 1.

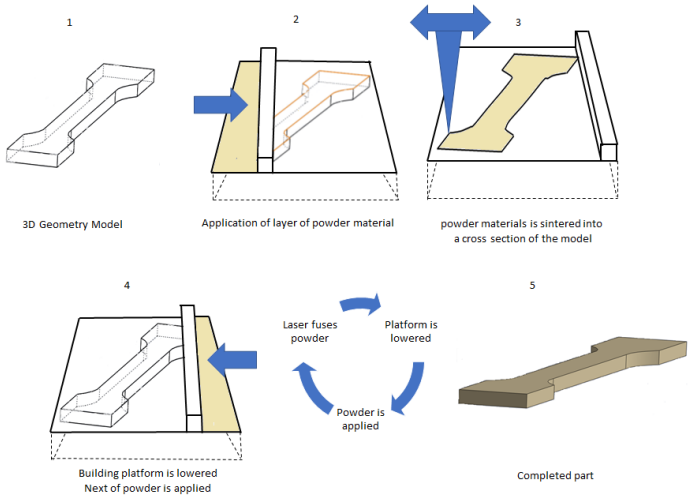


Figure 1: A schematic of the HT-SLS process used to produce the PEEK samples.

Sample geometries were produced in the x-y plane and were based on the corresponding International Standard Organisation (ISO) standard (described for each test below). This allowed the mechanical properties of the samples to be evaluated against existing data for high performance polymers. However, it should be noted that the mechanical tests performed are based on standards for polymers processed by injection moulding as, currently; no standard for the mechanical testing of HT-SLS materials exists. Therefore, as roughness was a result of the manufacturing process, samples were tested 'as-produced', i.e. neither polished nor machined to fit roughness tolerances suggested in the ISO standards.

2.1. Physical Properties

A JEOL 6060 Scanning Electron Microscope (SEM) was used to observe the surface characteristics of the HT-SLS material. The sample was gold-coated. The topography of the surface was measured using an Alicona G5 InfiniteFocus (Alicona, Raaba, Austria) using 10× magnification.

A mercury porosimetry was used to examine sub-surface pores in the material. The mercury porosimetry analysis used the intrusion of mercury into a porous structure under controlled pressures to determine sample properties such as pore size distributions, total pore volume, median pore diameter, and sample densities (bulk and skeletal). In this study, a Micrometrics AutoPore IV was used allowing theoretical pore diameters from 6 nm to 360 µm to be detected. Mercury intrusion into the sample was analysed using the Washburn equation (Equation 1) in order to determine the pore diameter distribution, assuming cylindrical pore geometry (15):

$$D = -\frac{4 \cdot \gamma}{P} \cdot cos\theta \qquad \textbf{Equation 1}$$

where *D* is pore diameter, *P* is the applied pressure, γ is the surface tension of mercury at 20 °C, which was assumed to be 0.485 N/m, and θ is the contact angle between the mercury and the solid, which was assumed to be 130 degrees (15).

2.2. Mechanical Properties

The tensile, flexural, fractural and compressive responses of EOS PEEK HP3 were investigated following the appropriate ISO guidelines for polymeric materials,

2.2.1. Tensile Testing

(EN ISO 527-1:2012 and EN ISO 527-2:2012).

In this test, five tensile tests were run following ISO 527-2/1A/1; this corresponds to an extension rate of 1mm/min and a strain rate of approximately 1%/min assuming uniform

deformation. The specimen dimensions were determined according to **EN ISO 527-2:2012** Plastics - Determination of tensile properties.

2.2.2. Flexural Testing

BS EN ISO 178:2010 describes a three-point-bending method for determining the flexural strength and flexural modulus rigid and semi-rigid plastics. In this test, five samples were tested at a strain rate of approximately 1% min^{-1}.

2.2.3. Fracture Testing

BS ISO 13586:2000 suggests a test method to establish the fracture toughness of the samples. Test specimens had a notch manufactured into them; allowing a sharp edge to be produced, post production, by sliding a razor blade across the notch. The test sample was then loaded to failure. Three test specimens were tested to ensure test consistency. The test equipment conforms to **BS EN ISO 178:2010** for the determination of the flexural properties of a polymer under three-point bending.

2.2.4. Compressive Testing

BS EN ISO 604 (2003) describes a test method for determining the compressive strength and compressive modulus of a polymer. In the measurement of compressive modulus and strength, two different geometries and operating conditions were chosen:

- For measurements of compressive modulus, five type A right prism compressive specimens (Length, l: 50 ± 2 mm, width, b: 10 ± 0.2 mm, thickness, h: 4 ± 0.2 mm) were tested at ISO 604/A/1, corresponding to a compression rate of 1mm min^{-1}.
- For measurements of compressive strength, five type B right prism compressive specimens (Length, l: 10± 0.2 mm, width, b: 10 ± 0.2 mm, thickness, h: 4 ± 0.2 mm) were tested at ISO 604/B/5, corresponding to a compression rate of 5mm min^{-1}.

2.3. Thermal Properties

Two methods of thermal characterisation were used in this study: Differential Scanning Calorimetry (DSC) and Dynamical Mechanical Thermal Analysis (DMTA). A Perkin Elmer DSC 7 unit (PerkinElmer Inc., Coventry, UK) was used to measure variation in relative heat flow of the material with temperature and provided an accurate method of determining the glass transition temperature (Tg), and melting temperature (Tm) of the material.

DMTA was used to characterise the viscoelastic or time-dependent behaviour of samples.

By applying a sinusoidal load to the sample, the time-dependence of the resultant strain was measured with respect to the applied stress. This allowed the in-phase storage modulus (E')

and the out-of-phase loss modulus (E”) to be measured. Their ratio E”/E’ defines the loss tangent (tan δ).

In this study DMTA tests were performed using a Rheometric Scientific DMTA MK III (Rheometric Scientific, Loughborough, UK). The experiments were performed in a dual-cantilever arrangement in three-point-bending for a range of oscillation frequencies. The samples were heated from 100 to 250 °C at a heating rate of 0.5 °C/min and the displacement was kept constant at 20 µm to ensure measurements were performed for the linear viscoelastic region.

3.0. Results and Discussion

3.1. Physical Property Characterisation

Figure 2 shows the surface of the EOS PEEK HP3 laser sintered material. The higher magnification image shows individual particles indicating that the surface was only partially sintered. This will affect the topography and surface roughness.

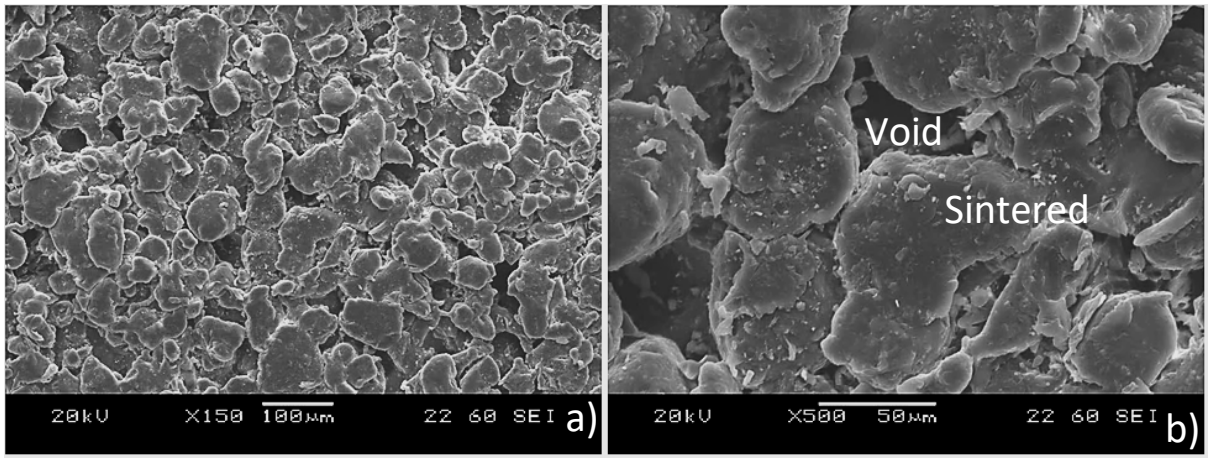


Figure 2: SEM micrographs of, a) the as-produced surface of EOS PEEK HP3 b) detailed showing partially sintered material

Laser sintering is known to give a porous surface due to the partial sintering of the powder on the edges of the component geometry (14), however, this can be minimised through careful parameter selection. The nano-scale porosity through the sintered material was investigated. The EOS samples tested were 4.36% porous and the average pore diameter was 20.4 nm. Table 1 summarises the results of the porosity testing

Table 1: Porosity results for EOS PEEK HP3 samples

Total Intrusion Volume	0.035	mL/g
Median Pore Diameter (Volume)	47.6	nm
Median Pore Diameter (Area)	9.5	nm
Average Pore Diameter (4V/A)	20.4	nm
Bulk Density at 0.51 psia	1.25	g/mL

Apparent (skeletal) Density	1.31±0.2	g/mL
Porosity	4.36	%

Figure 3 shows the dispersion of values of pore diameter for the EOS samples. It can be seen that the material porosity consisted of sub-100 nm pores, which were mainly 10 nm or smaller. This may have an effect on the fatigue properties of the material. The dotted line represents the minimum measurable pore diameter and is a limitation of the testing equipment. Hooreweder *et al*. compared the material performance of laser sintered and injection moulded polyamide and found that injection-moulded and selective-laser-sintered samples had similar fatigue properties despite the presence of pores in the sintered samples. They attributed this to the high molecular mobility, resulting from the high local cyclic stresses near the pores or initial cracks, increasing the inner material temperature and thus delaying fracture (7). Porosity has also been measured by microtomography (µCT). Beard *et al.* used µCT techniques to analyse the sub-surface properties of EOS PEEK HP3. They concluded that the parts were fully dense throughout (14). The subjective nature of analysing µCT images, especially when attempting to see nano-scale porosity, limits its applicability to only measuring large-scale pores, as small-scale porosity cannot be seen.

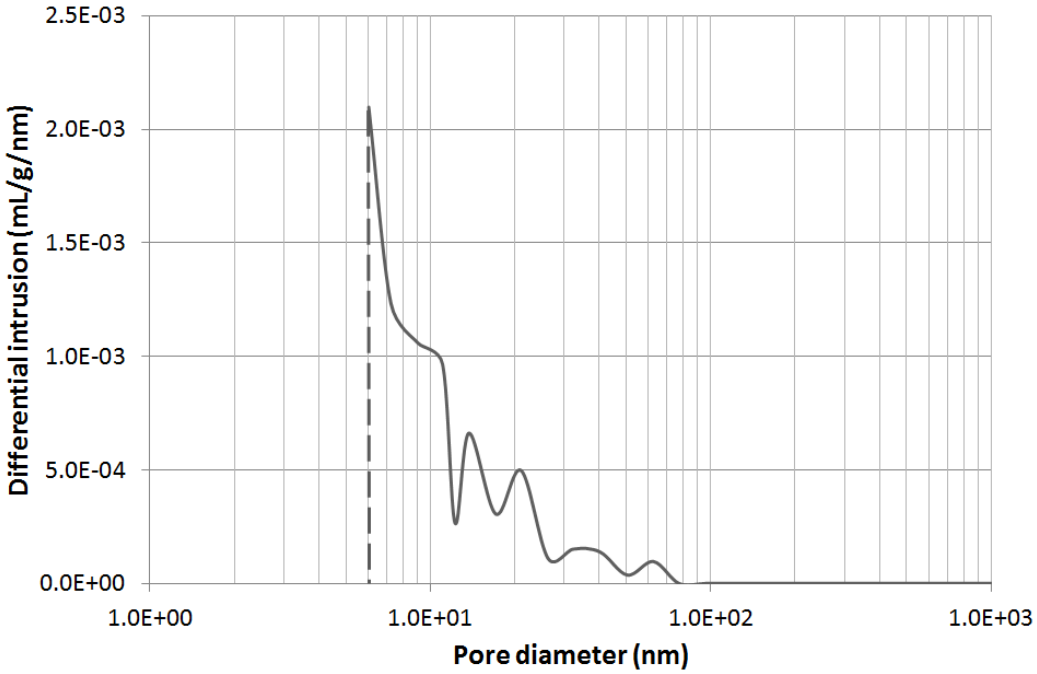


Figure 3: Differential intrusion (defined as the incremental specific intrusion volume divided by the difference in diameter over which the volume increment is calculated) vs. Pore diameter for EOS PEEK HP3

The roughness characteristics of the laser-sintered surface are dependent on the orientation of the component produced. The vertical surface roughness is generally higher than the horizontal due to the manufacturing process. Table 2 quantifies the two 3D roughness profiles shown in Figure 4.

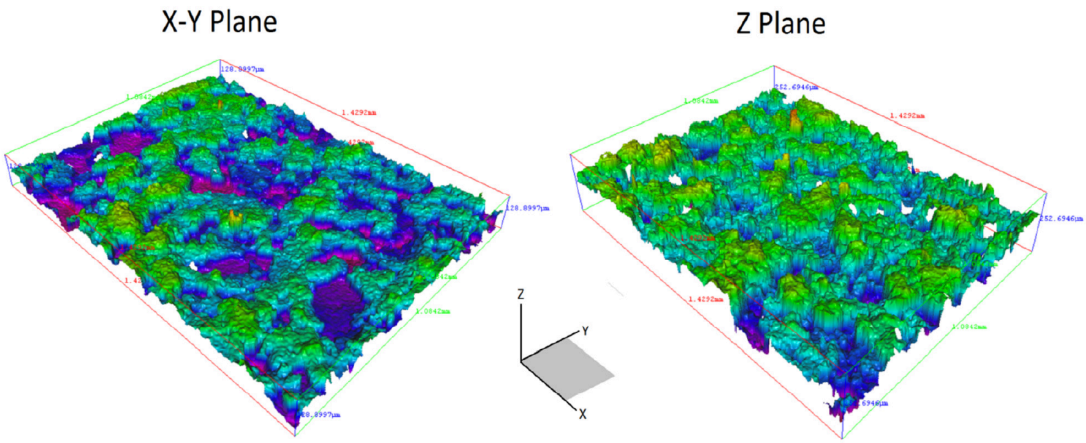


Figure 4: Surface roughness profile for EOS PEEK HP3

As expected, the roughness of the surface on the z-axis is significantly more than on the x-y axis. However, it is interesting to note the differences in skewness and kurtosis. Both surfaces have negative skewness, due to the partially-sintered outer surface of the material. However, the x-y axis kurtosis shows rounded asperities whereas the z-axis shows sharper asperities introducing an anisotropic surface topography. This is a consequence of the manufacturing process and is likely to have a significant impact on the mechanical and tribological properties of the surfaces. Thus, due to the high roughness, and the impact this may have on wear mechanisms and debris production, some surfaces may require additional surface finishing (such as polishing or lapping) following manufacture to remove the potential anisotropic surface topography and maintain uniform wear over the entire surface.

Table 2: Measured Roughness of EOS PEEK HP3 parts

Property	X-Y Axis	Z Axis
Sa (Average asperity height)	16.37 µm	23.87 µm
Sq (Root-Mean-Square height)	19.86 µm	29.75 µm
Ssk (Skewness)	-0.32	-0.44
Sku (Kurtosis)	2.40	3.15

3.2. Mechanical Property Characterisation

From tensile testing, the EOS PEEK HP3 samples were found to have a tensile modulus of 2.76 ± 0.15 GPa, a tensile strength of 88.7 ± 1.5 MPa, and an elongation to break 4.2 ± 0.2 %. The tensile specimens showed very little plastic deformation, with a sudden fracture occurring; typical of brittle materials. Figure 5 shows the fracture surfaces of the tensile test specimen.

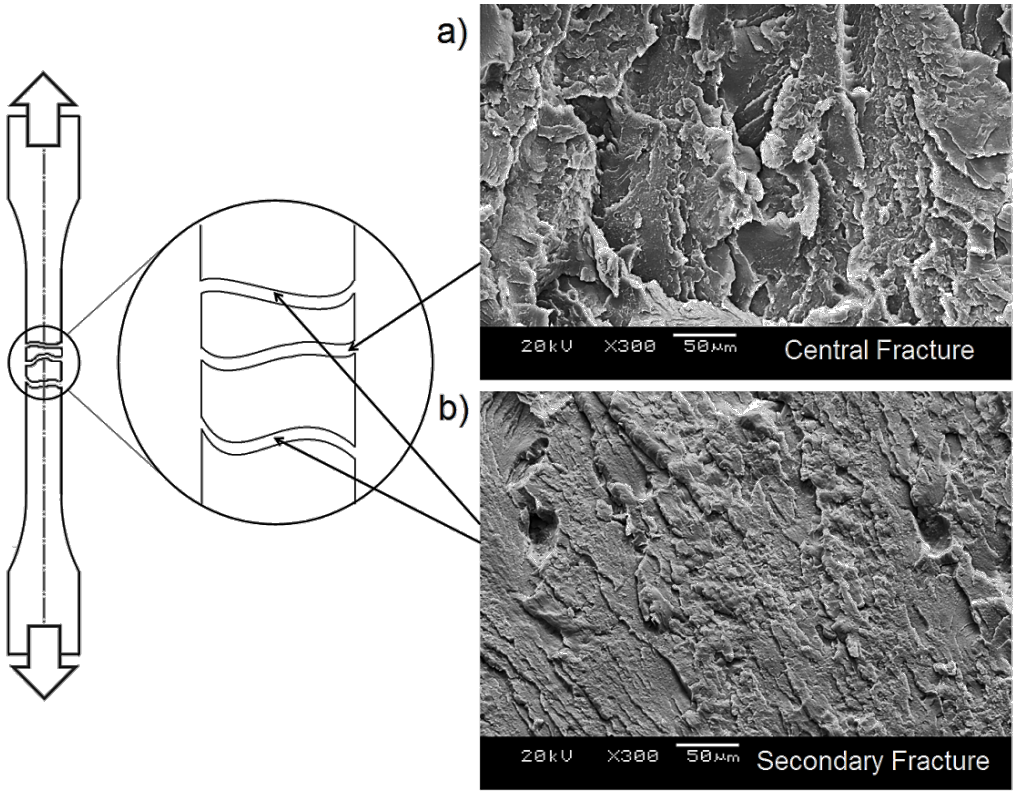


Figure 5: Fracture surfaces from tensile testing; a) central fracture, b) secondary fracture

The tensile samples each had a central fracture (Figure 5a) and surrounding secondary fractures (Figure 5b); these secondary fractures are attributed to the stress wave travelling through the material after the initial failure. This is supported by observations of the failed surfaces; it can be seen that the central fracture was progressive and showed a small amount of plasticity in failure; however, the secondary failures were typical brittle failures, showing very smooth failure planes with no identifiable necking of the sample.

Flexural testing showed a similar fracture response to that in tensile testing, but the flexural modulus and strength were significantly higher at 3.26 ± 0.7 GPa and 123.0 ± 2.5 MPa respectively. The initial fracture surface was similar in appearance, although, there were no secondary fractures resulting from the initial failure. Figure 6 shows the fractured surface. Two regions can be identified from the surface; an initial region of progressive failure, and a region of fast fracture. The layers of sintered material (approximately 120 µm) can be clearly seen in Figure 6a.

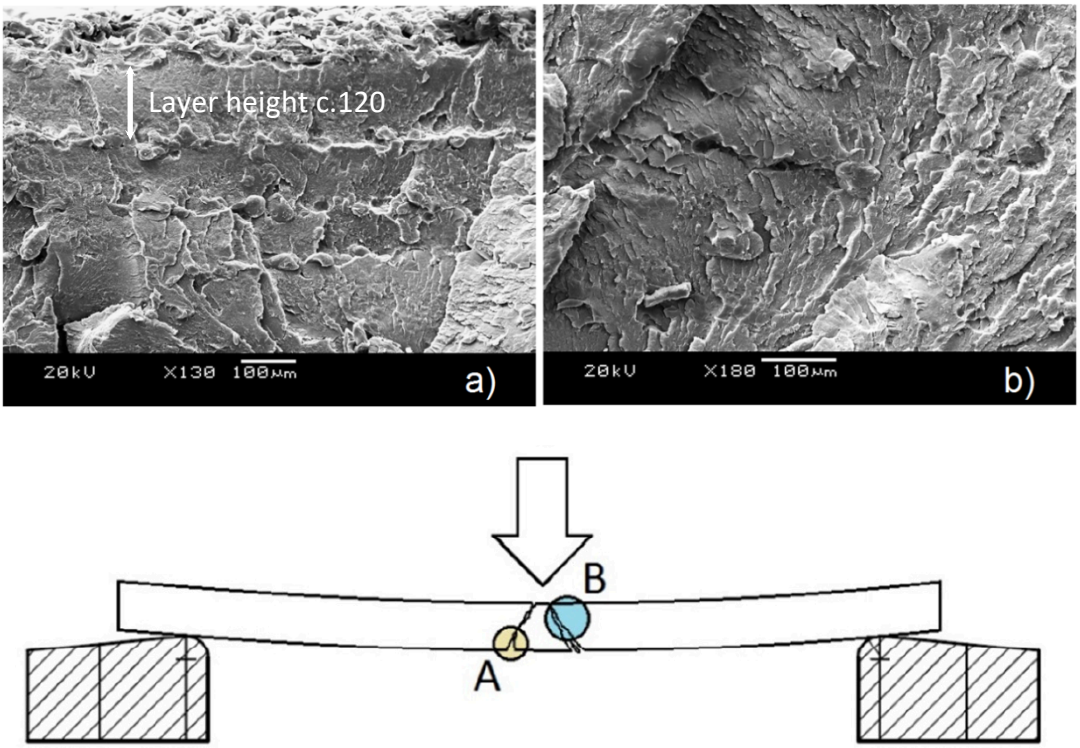


Figure 6: flexural failure; a) progressive fracture of layers b) fast fracture

Fracture toughness (K_{IC}) was calculated according to BS ISO 13586:200 as 1.40 ± 0.2 MPa/m^2. This is comparatively low when compared with that for injection moulded PEEK ranging between 5.4-7.5 MPa/m^2 (Vitrex, 2013). Figure 7 shows the fracture surface. Due to the initiation of the crack in the as-processed sample the fracture was mainly brittle in nature.

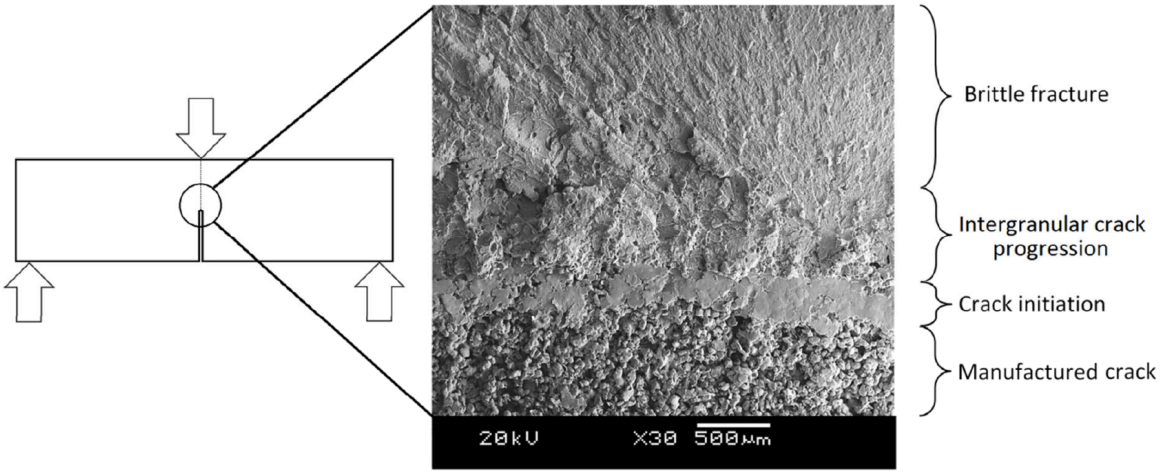


Figure 7: Fractured surface showing regions of failure (Note: crack propagation is from bottom to top)

Compressive testing was done in two stages; the compressive modulus was determined to be 610 ± 15 MPa and the compressive strength to be 184 ± 15 MPa. The compressive strength samples showed severe delamination following a period of deformation (Figure 8), indicating that anisotropy of the sample had a significant effect on failure characteristics.

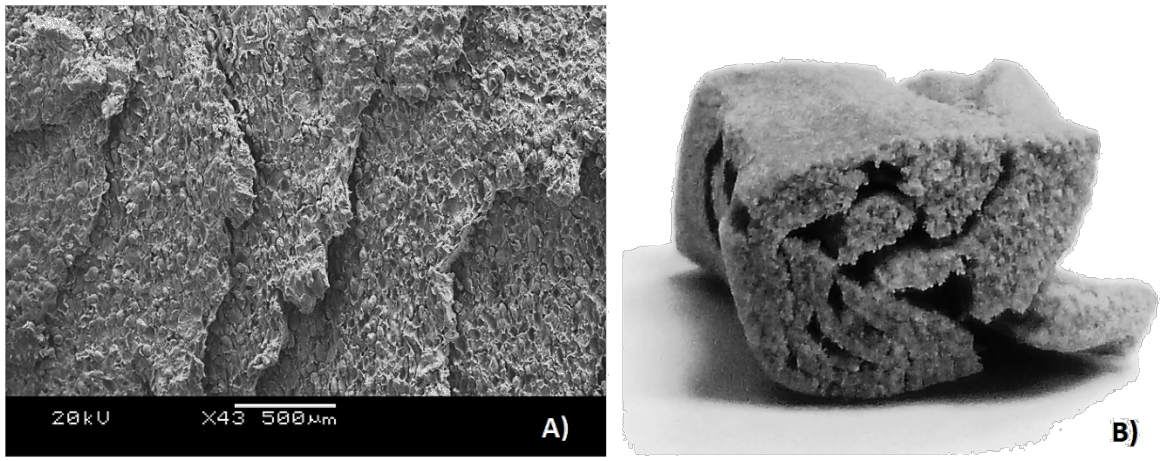


Figure 8: Compressive EOS PEEK HP3 sample delamination showing, a) the fractured surface and b) the compressive strength sample following test.

The delamination in the compressive samples occurred in two stages: firstly, it is most likely that void nucleation and coalescence at the boundary between the sintered layers resulted in crack propagation in the sample. Following this, progressive delamination between the layers occurred as the stress induced between these layers was increased. Finally, a period of fast fracture occurred where the surfaces were ruptured.

Figure 9a shows two regions of failure within one of the compressive samples. The failure region, shown in greater magnification in Figure 9b is created as the sample is compressed. Following delamination, the fracture surfaces are free to move and the loaded surfaces within the sample move against the failed surface forming the smoothed topography shown in Figure 9c.

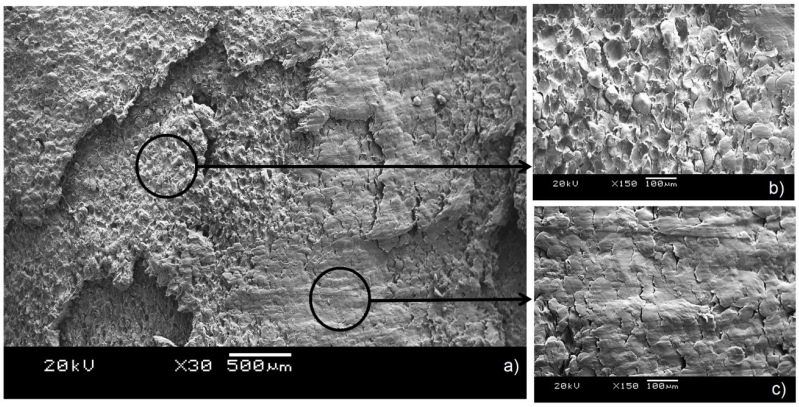


Figure 9: Compressive failure surfaces showing a) regions of failure, b) partially sintered material, c) worn path due to failure.

The failure surface in the compressive strength samples (Figure 9b) shows a significant amount of partially sintered material. Particles can be clearly identified and are approximately 60µm in diameter; correlating with the reported particle size for EOS PEEK HP3 powder (16). This is an issue as the inter-granular strength at the interface between the layers is not sufficient to hold together the material under high loads; as shown by the failure surface. Thus, it is necessary to refine the material processing parameters to allow for a fully sintered material to be produced. This should increase the fracture toughness and also the mechanical strength of the material.

The mechanical performance of EOS PEEK HP3 is dependent on the mechanism of loading. The response of the material is predominantly brittle, with failure surfaces showing delamination, sharp fracture planes and surface tearing.

Partially sintered material is still identifiable in the finished material and can be seen to significantly affect the mode of failure. When the partially sintered material is located on the surface of the component, the porosity provides an exciting potential for lubricant retention similar to surface texturing. Modifying the surface of engineering surfaces, through the formation of micro-surface structural forms such as dimples has been shown to significantly improve the load carry capacity and tribological properties (Hammouti et *al*. 2015). The surface porosity may offer similar benefits that may be tuneable for particular applications.

3.3. Thermal Property Characterisation

As the sample passes through Tg, the rate of energy absorption goes through a maximum; resulting in peaks in the loss modulus (E”) and tan δ curves. The glass transition can be characterised based on the three DMTA events. For a frequency of 1Hz, the Tg values are 170.2 °C (storage modulus, E' onset), 178.6 °C (E" peak) and 186.8 °C (tan δ peak), as shown in Figure 10.

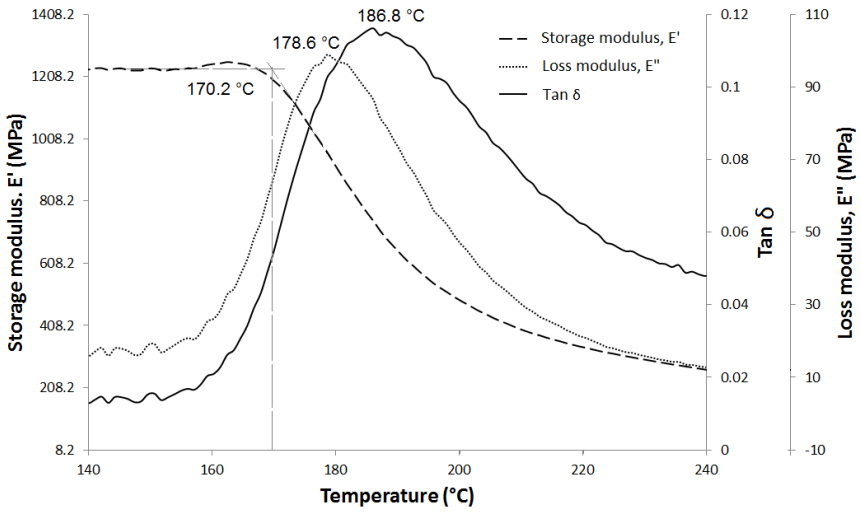


Figure 10: Glass transition temperature of EOS PEEK HP3 as determined by DMTA using three methods, for 1Hz.

The E' (storage modulus) onset defines the temperature at which the material strength will begin to decrease, such that the material may no longer be able to bear a load without significant deformation. The peak in the loss modulus (E") represents the temperature at which the material undergoes the maximum change in polymer mobility, which corresponds to Tg, and the loss tangent (tan δ) peak describes the damping characteristics of a material.

Figure 11 shows the progression of storage modulus and tan δ with frequency. It can be seen that as the frequency increases, there is a slight decrease in the intensity of the tan δ, a broadening of the tan delta peak, and a decrease of the slope of the storage modulus curve in the region of the transition.

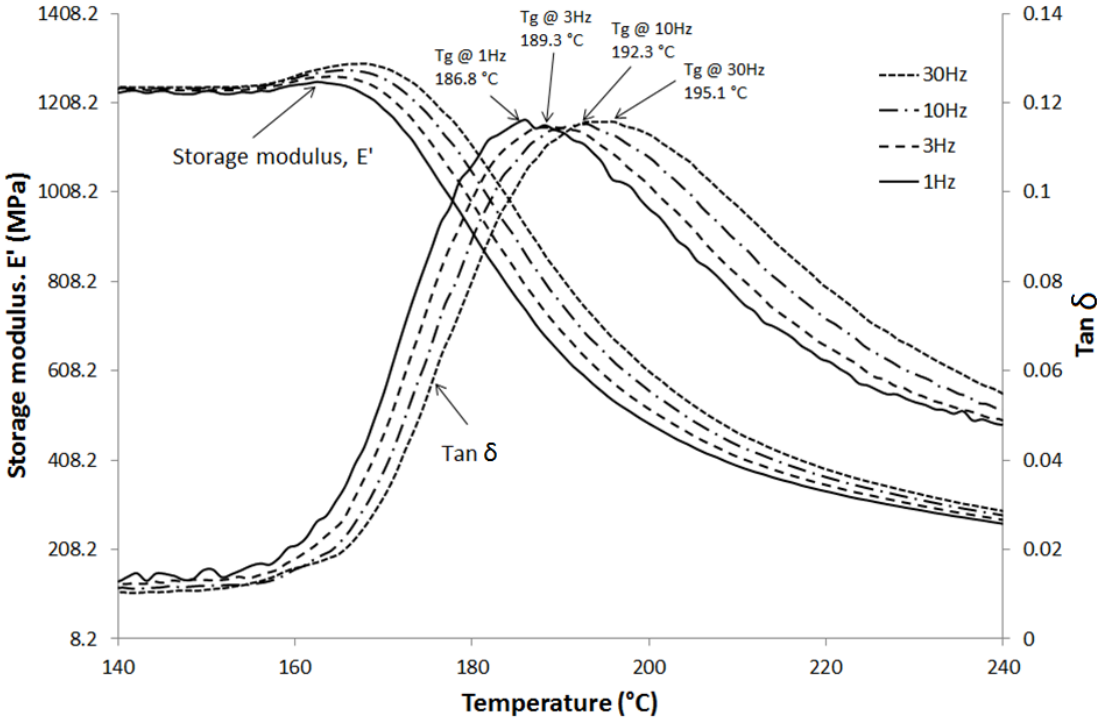


Figure 11: DMTA output, showing the progression of storage modulus and tan δ with frequency for EOS PEEK HP3.

Table 3 shows the Tg determined using the three different methods from the DTMA analysis. These values demonstrate the applicability of the material for high temperature mechanical application. The dynamic glass transition temperatures are a significant improvement on the values typically associated with laser sintered polymers (6).

Table 3: Tg of EOS PEEK HP3 based on the sinusoidal oscillation frequency; DMTA methods

	Oscillation Frequency			
Method	**1 Hz**	**3 Hz**	**10 Hz**	**30 Hz**
tan δ	186.8 °C	189.3 °C	192.3 °C	195.1 °C
E" peak	178.6 °C	181.5 °C	183.4 °C	185.3 °C
E' onset	170.2 °C	172.3 °C	173.8 °C	177.1 °C

The physical and mechanical properties of semi-crystalline polymers are highly dependent on their degree of crystallinity. To characterise the crystallinity of EOS PEEK HP3 DSC was used. Assuming the enthalpy of fusion of the pure crystalline phase of PEEK to be 130J/g (17), it was found that the EOS PEEK HP3 had a crystallinity of 35%. This is comparable with typical values for high performance injection moulded polymers, making it much stronger, stiffer and denser than previous laser sintered material (Katti and Schultz, 1982).

From the DSC analysis, for a heating rate of 10 °C/min, the glass transition and melt temperature were found to be 160°C and 398°C respectively. These values are both significantly higher than those typically associated with laser-sintered polymers (18). The thermal characteristics of EOS PEEK HP3 are far superior to previous laser sintered polymers indicating its suitability for use in high temperature mechanical applications.

3.4. Materials Property Comparisons

The use of high temperature semi-crystalline materials in HT-SLS has significant benefits in terms of component development and manufacture. However, it is necessary to quantify the thermal and mechanical properties against those of similar materials to validate its use.

Table 4 shows a comparison of the material properties of EOS PEEK HP3, the reported properties for injection moulded PEEK 450G and selectively laser sintered PA 2201/PA12.

- PEEK 450G is the general grade injection moulded PEEK developed by Victrex (19). It is widely regarded as one of the highest performance mechanical polymers.
- PA 2201/PA12 is an unreinforced, laser sintered polyamide developed by EOS for the EOSINT P (20). This material is typically applied to components exposed to high mechanical and thermal loads.

Temperature has thus far limited the application of laser sintered materials such as PA 2201/PA12. However, it can be seen that EOS PEEK HP3 has a melting point 200 °C higher than PA 2201/PA12, and a glass transition temperature considerably higher (defined by the peak in the tan δ curve at 1Hz). In addition, it can be seen that the tensile and flexural properties of PEEK 450G and EOS PEEK HP3 are both higher than those reported for PA 2201/PA12. The low elongation to break of EOS PEEK HP3 shows that the material is more brittle than PEEK 450G and PA 2201/PA12; this is to be expected as the porosity in the material will encourage fracture.

Table 4: Mechanical Properties summary (Sepe, 1998; Ajoku *et al.*, 2006; Rae, Brown and Orler, 2007; Salmoria *et al.*, 2008; EOS GmbH, 2014a, 2014b; Victrex PLC, 2014)

Material	EOS PEEK HP3	PEEK 450G	PA2201/PA12 (SLS)
Tensile Modulus (EN ISO 527)	2.76 ± 0.15 GPa	3.7 GPa	1.70 ± 0.15 GPa
Tensile Strength (EN ISO 527)	88.7 ± 1.5 MPa	100 MPa	45 ± 3 MPa
Elongation at Break (EN ISO 527)	4.2 ± 0.2 %	34 %	20 ± 5 %
Compressive Modulus	0.61 ± 15 GPa	3.45 GPa	0.69 GPa
Compressive Strength	184 ± 15 MPa	125 MPa	53 ± 15 MPa
Flexural Modulus (EN ISO 178)	3.26 ± 0.7 GPa	4.1 GPa	1.24 ± 0.13 GPa
Glass Transition Temperature (DSC)	174.5 °C	143 °C	52 °C
Fracture Toughness, K_{IC}	1.40 ± 0.2 MPa/m^2	5.4 - 7.5 MPa/m^2	-
Glass Transition Temperature (DMTA, tan δ)			
@1Hz	186.8 °C	165.8 °C	48 °C
@3Hz	189.3 °C	-	-
@10Hz	192.3 °C	-	-
@30Hz	195.1 °C	-	-
Density: Bulk (DIN 53466)	0.43 ± 0.01 g/cc	1.30 g/cc	0.44 ± 0.01 g/cc
Density: Laser sintered part (DIN EN ISO 1183)	1.307 ± 0.01 g/cc	N/A	0.930 ± 0.01 g/cc
Porosity (BS ISO 15901)	4.359 %	N/A	7 %
Melting point (DSC)	372 °C	343 °C	172-180 °C
Crystallinity (DSC)	35.4 %	30 %	-

Overall, there are small discrepancies between the measured parameters in this study and the reported values for EOS PEEK HP3. This is considered to be a result of variation in manufacturing parameters as well as the significance of the anisotropy of the samples; as the samples were all produced in the x-y plane, the effect of material anisotropy was not investigated but has been shown to have a significant effect. The thermal and mechanical properties of EOS PEEK HP3 are far superior to previous selectively laser sintered polymers and closer to that of reinforced, higher performance injection moulded PEEK blends. This demonstrates the applicability of this material for high load, high temperature applications. Figure 12 shows the improvement in material properties using EOS PEEK HP3 over conventional SLS materials. The thermal and mechanical properties of EOS PEEK HP3 are far superior to previous selectively laser sintered polymers and closer to that of higher performance injection moulded PEEK blends.

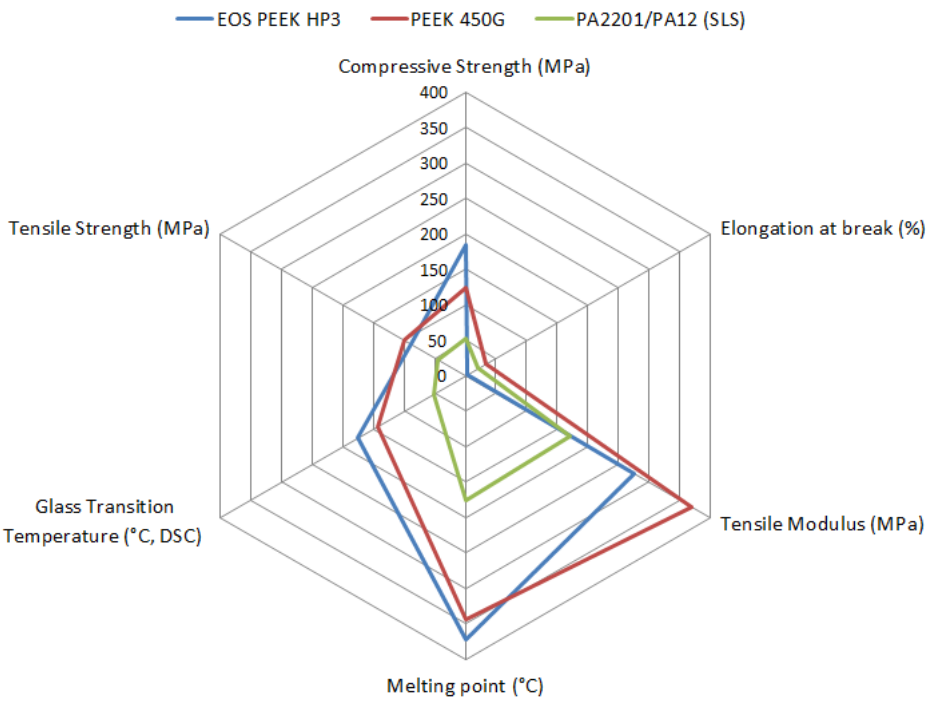


Figure 12: The comparative properties of EOS PEEK HP3 against injection moulded PEEK 450G and PA2201/ PA12 produce using selective lasers sintering.

4.0 Conclusions

Traditionally, the properties of a laser-sintered material would be expected to be significantly worse than those of high-performance injection-moulded polymers. However, it has been shown that the performance of EOS PEEK HP3 is significantly higher than for previous laser-sintered materials and comparable with high performance injection-moulded materials.

The failure mechanisms observed vary depending on the method of loading; secondary fractures were observed in tensile testing whereas delamination of the material could be seen at higher loads in compressive testing. The surface of the failed compressive strength samples showed partially sintered material, this should be evaluated to improve the material properties and refine processing parameters, as this was not immediately apparent from tensile failures.

Overall, the use of EOS PEEK HP3 for impact loading is not recommended as the brittle tendencies may lead to sudden failure. However, design optimisation methods to allow for the distribution of load across a surface are recommended to improve the systemic performance.

As with any material, the specific design requirements may vary. Thus, further analysis to replicate the precise operational conditions may be needed. However, this paper outlines the fundamental mechanical, thermal and physical characteristics of the material and can be used to give an indication of the suitability of HT-SLS materials for mechanical applications.

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